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<p>(21) International Application Number: PCT/EP96/02586</p> <p>(22) International Filing Date: 14 June 1996 (14.06.96)</p> <p>(30) Priority Data: 95304401.3 22 June 1995 (22.06.95) EP <i>(34) Countries for which the regional or international application was filed:</i> GB et al.</p> <p>(71) Applicant (for all designated States except AU BB CA GB IE KE LK LS MN MW NZ SD SG SZ TT UG): UNILEVER N.V. [NL/NL]; Weena 455, NL-3013 AL Rotterdam (NL).</p> <p>(71) Applicant (for AU BB CA GB IE KE LK LS MN MW NZ SD SG SZ TT UG only): UNILEVER PLC (GB/GB); Unilever House, Blackfriars, London EC4P 4BQ (GB).</p> <p>(72) Inventors: VAN DIJK, Willem, Robert; Van den Bergstraat 36, NL-3263 EB Oud Beyerland (NL). OUWENDIJK, Marya; Albert Schweitzerlaan 51, NL-3223 WE Hellevoetsluis (NL). HALL, Peter, John; 237 Spital Road, Bromborough, Wirral, Merseyside L62 2AF (GB).</p>		<p>(81) Designated States: AL, AM, AT, AU, AZ, BB, BG, BR, BY, CA, CH, CN, CZ, DE, DK, EE, ES, FI, GB, GE, HU, IL, IS, JP, KE, KG, KP, KR, KZ, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, TJ, TM, TR, TT, UA, UG, UZ, VN, ARIPO patent (KE, LS, MW, SD, SZ, UG), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TO).</p> <p>Published <i>With international search report.</i></p>
(54) Title: ENZYMATIC COMPOSITION		
(57) Abstract		
<p>Enzymatic compositions with improved storage stability of the enzymes contained therein are obtained by including an enzyme stabiliser, preferably by way of a particular process.</p>		

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ENZYMATIC COMPOSITION

Technical field

The present invention relates to a enzymatic composition with improved storage stability of the enzymes contained therein.

Background & Prior art

It is well known in the art that enzymes can lose their activity with time when included in an aqueous liquid detergent composition, and various proposals have already been made to retard that loss of activity by including in such compositions an enzyme-stabilizing system. Various enzyme stabilisers have been suggested in the art for inclusion in liquid detergent compositions, e.g. polyols (e.g. glycerol), borax (preferably in combination with glycerol), calcium ions, alcohols, low molecular weight carboxylates (formate, acetate, propionate, etc.) and polymers (e.g. poly-vinyl-pyrrolidone).

20 We have surprisingly found that inclusion of a certain class of compounds in such aqueous enzymatic liquid detergent compositions retards the loss of enzyme activity.

Statement of the Invention

25 We have found that enzyme stability can be improved by using the class of compounds that embraces the group of lignin compounds.

Therefore, the present invention relates to an enzymatic detergent composition with an improved storage stability of enzyme material contained therein, the improved storage stability being obtained by the inclusion in the composition of a lignin compound.

Description of the Invention

Lignin compounds are mixtures of components and is usually referred to as a polymer which contains, amongst others, phenylpropane units. Lignin compounds can be prepared from the chemical pulping of hard- and softwoods. Lignin compounds have been found to be very effective compounds according to the present invention. There are various lignin compounds which are preferred enzyme stabilisers according to the invention, including Lignosulphonates, Kraft lignins and Oxylignins. All these compounds are considered lignin compounds. These compounds may be prepared from Lignin by various ways, including:

- 1) treatment with hot (acid) solution of calcium bisulphite which generates Lignosulphonates. The Lignin undergoes a sulphonation and a hydrolysatation process under the influence of sulphite.
- 2) treatment with hot alkaline (pH 13-14) solution of sodium sulphate generates Kraft Lignins, which may subsequently be modified in various ways, e.g. sulphonated, methylated, carboxylated and/or fractionated.
- 3) reducing the sulphur content of lignosulphonate raw material and optionally applying condensation, cleavage and/or rearrangement, to reduce the number of sulphonic and methoxyl groups and to increase the number of functional phenolic, hydroxyl and carboxylic groups generates Oxylignins.

Further variations to Lignin or any of its derivatives may be made by varying the kind of cation (Na^+ , K^+ , Ca^{2+} , Mg^{2+} , NH_4^+ , the degree of sulphonation and/or the average molecular size.

Examples of lignin derivatives that have been found useful are Borresperse NA, Borresperse CA, Kelig FS, Maracarb N-1, Marasperse N-22, Marasperse N-3, Norlig BD, Norlig 415, Ufoxane 2, Ufoxane 3A, Maracell 3A, Vanisperse CB, 5 Ultrazine NA, Ultrazine CA (all ex Borregaard) and lignosulphonates ex Aldrich and ex Sigma as well as ex a number of pharmaceutical companies.

We have found that inclusion of lignin compounds 10 significantly retards the enzyme deactivation, and most surprisingly, lignin compounds are effective as stabiliser at low concentration. Consequently, lignin compounds are included in effective amounts in the composition, in particular in the range of 0.0001 to 10%, preferably 0.001 15 to 5%, more preferably at least 0.01 and more preferably at most 3% by weight of the composition.

Although the weight ratio between lignin compound and enzyme (as defined as the weight of the active enzyme 20 protein material, which does not include any additives that for example may be present in the enzyme preparations as supplied by the enzyme manufacturers) may be varied widely, as long as the enzyme is effectively stabilised, a weight ratio between 1000:1 and 1:10 has been found to be 25 preferred, more preferably lower than 500:1, most preferably lower than 100:1, in particular lower than 50:1, whereas it is more preferred to have a weight ratio of higher than 1:5, most preferably higher than 1:3, in particular 1:2, more in particular 1:1.

30

Preferably, the molar ratio between lignin compound and enzyme is from 0.1 to 10,000, more preferably at least 1 and at most 5,000, most preferably at least 2.

It will be understood that presence of other enzyme stabilising systems is not excluded in compositions according to the invention.

- 5 Lignin compounds have been described in the art for several applications.

GB-A-1,403,257 discloses use of lignin in enzyme preparations which may be included in solid compositions.

- 10 The enzyme preparations are purified by precipitating protease or α -amylase with a tanning agent or lignin, whereafter the solid enzyme preparation is filtered off.

- DE 23 54 791 discloses the use of lignosulfonates as
15 coating material for enzyme granules for use in powdered compositions.

- DD 237,522 discloses a process for cleaning an enzyme concentrated containing protease and/or by α -amylase by
20 precipitating undesired polution.

- Use of lignin preparations to inhibit enzyme activity at low pH in the human stomach is discussed in ZA 6803394 and in EUR J Pharmacol 41 (2) 1977 p 235-238; coden: EJPHAZ
25 ISSN: 0014-2999 [EMW].

- WO 94/19529 discloses a process for providing localized variation in the colour density of fabrics by using a cellulase enzyme and a polymeric agent.

30

The invention further relates to a liquid enzymatic composition comprising from one or more enzymes and one or more enzyme stabilisers, characterised in that the stabiliser comprises a water-soluble, cross-linked polymer

containing sulphonate-groups, preferably containing benzene units and more preferably containing phenylpropane units.

The enzymatic composition of the present invention contains as essential ingredients one or more enzymes, preferably at least including a proteolytic enzyme.

The enzymes that may be used in the present invention are proteases, amylases, lipases, cellulases and mixtures of one or more of these enzymes. Proteases are preferred enzymes for use in the present invention, as we have seen the best results with protease stabilisation.

Depending on the type of composition (i.e. diluted or concentrated enzyme composition) and, of course, whether the enzyme type is present at all, the enzymes preferably provide a proteolytic activity between 0.1 and 50 GU/mg, a lipolic activity between 0.005-100 LU/mg and an amylotic activity between 10^3 to 10^7 MU/kg, wherein GU, LU and MU units are well known in the art and have e.g. been defined in lines 8-14 of column 3 and lines 8-12 and 21-24 of column 4 of US 5,112,518.

Depending on the composition type, the level of active enzyme protein will be higher (up to 10%, preferably up to 5% by weight for concentrated enzyme preparations, e.g. as supplied by enzyme manufacturers) or lower (up to 3%, preferably up to 1.0%, although levels up to 0.5% or up to 0.1% or even as low as up to 0.05% are also suitable for more dilute systems, e.g. commercial liquid detergent compositions in which the concentrated enzyme preparations are used during production). The active enzyme protein level may be as low as 0.0001%, preferably at least 0.01% by weight of the composition. Again in more concentrated

enzyme preparations, the lower level will be higher, e.g. at least 0.5% by weight.

We have further found that combinations of enzymes
5 (especially when they include proteases) may be stabilised by using the invention. As compared to the composition without the stabiliser, they show strongly improved stability overall.

10 Preferably, detergent-active component is included and may be either soap, anionic, nonionic, cationic or zwitterionic detergents and mixtures of one or more of these detergent-active components. Preferably, nonionic detergent is used,
15 either as such or in admixture with a anionic detergent-active component. Usually, the total amount of detergent-active component(s) ranges from 5% to 70%, preferably from 10 to 40% by weight of the total composition.

Preferably, compositions according to the invention have an
20 ionic strength and contain electrolyte material.

Preferably, electrolyte material is selected from the group consisting of phosphate, phosphonate, borate, carboxylates (e.g. citrate, NTA and succinate such as C12
alk(en)ylsuccinate), carbonate, sulphate and chloride.
25 Preferably, the electrolyte material is present at a level of at least 1%, more preferably at least 2%, most preferably at least 3%, in particular at least 5%, e.g. at least 10% by weight of the composition. Suitable levels are at least 15% by weight of the composition. Preferably, the
30 composition comprises less than 25% by weight of electrolyte material.

The composition may furthermore contain other optional ingredients such as perfumes, colouring materials, soil-
35 suspending agents, other enzyme-stabilising agents,

builder, bleaching agents, bleach precursors, hydrotropes, solvents, suspending agents, suds suppressors, polymers (e.g. for oily soil or particulate soil removal or as anti-dye transfer agent), fluorescers, etc.

5

The enzymatic composition may be in the powdered form, but is preferably in the liquid form. The composition may be an isotropic or a structured liquid. Structured liquids (i.e. containing lamellar droplets of surfactant material) are
10 the most preferred liquids.

Preferably, liquids according to the present invention are prepared by mixing an enzyme preparation and one or more enzyme stabilisers, wherein the enzyme stabiliser comprises
15 lignin compounds.

Preferably, the pH of the liquid formulations according to the present invention is higher than 4, more preferably higher than 5, most preferably higher than 5.5 and preferably lower than 11, more preferably lower than 10,
20 most preferably 9.0 or lower.

To improve the enzyme stability even further, the lignin compound is preferably brought in contact with the enzyme in a form in which the lignin is at least partially
25 dissolved. This may be done in various ways, including choosing a certain order of addition that results in this effect. A premix of enzyme and lignin can be made which is then mixed with the other ingredients or lignin is added in the form of a solution, preferably in the form of a
30 solution in a solvent, e.g. selected from alcohols and/or water. Examples of suitable solvent systems are water and a 25% propyleneglycol solution.

The invention will now be illustrated by way the following
35 non-limiting examples.

EXAMPLESExample 1

The following formulation 1 was prepared:

5

<u>Ingredients</u>	<u>% by weight</u>
LAS (Na salt)	23
Nonionic*	10
Citrate (Na salt)	17
10 Polymer material**	1.0
Savinase 16.0L (ex NOVO)	0.38%
Minors	0.25
Water	to 100%

15 * Nonionic is an ethoxylated alcohol.

** (as 100%) Polymer A11 as described in EP 346,995.

The protease stability at 37°C was measured in the presence of various levels of sodium ligno-sulphonate. The following
20 results were obtained after 10 days.

	<u>% by weight</u> Ultrazine NA • ...	<u>% residual</u> activity
25	0	25
	0.005	52
	0.010	63
	0.015	68
	0.025	80
30	0.050	89
	0.100	82

*** a sodium lignosulphonate, ex Borregaard, added on top of formulation in powdered form.

It can be clearly seen that the lignin compound has good enzyme stabilising properties, even at very low concentrations.

5

Example 2

Lipolase® (100L, ex NOVO) was added to formulation 1 of Example 1 at a level of 0.2% and the lipase activity was determined after 10 days storage at 37°C.

10

% by weight Ultrazine NA* ***	% residual activity
-	3
0.005	3
0.010	5
0.015	5
0.025	38
0.050	50
0.100	80#

15

20

*** a sodium lignosulphonate, ex Borregaard, added on top of formulation in powdered form.

extrapolated from 7 days' stability data.

25

It can be clearly seen that the lignin compound has good lipolase stabilising properties in the presence of protease.

30

Example 3

The following liquid formulation 2 was prepared by neutralising a premix of the detergent active material, mixing in the builder material and the minors. Enzyme

material was added as last ingredient. Stabiliser (if any) was post-dosed.

	<u>Ingredients</u>	<u>% by weight</u>
5	Anionic	16.5
	Nonionic	4.5
	Oleic acid	4.5
	Citric acid	8.2
	Zeolite	15.0
10	KOH	10.3
	Polymer*	1.0
	Protease**	0.38
	Lipase ***	0.2
	Minors	0.9
15	Water	to 100

pH liquid 8.5

* Polymer A11 of EP 346995

20 ** Protease is Savinase 16.0L (ex Novo)

*** Lipase is Lipolase 100L (ex Novo)

The enzymatic activities in the liquid after 28 days of storage at 37°C was as follows when Ultrazine NA was added
25 in the form of a solution in 25% propyleneglycol solution:

	% by Ultrazine	% Residual protease act. (no lipase)	% Residual protease act. (with lipase)	% Residual lipase act. (with prot.)
30	NA			
	0	30	24	0
	0.025	58	63	22
	0.05	75	73	43
	0.1	79	80	53

35

The enzymatic activities in the liquid after 28 days of storage at 37°C was as follows when Ultrazine NA was added in solid form:

5	% by	% Residual	% Residual	% Residual
	Ultrazine	protease act.	prot. act.	lipase act.
	NA	(no lipase)	(with lipase)	(with prot.)
	0	30	24	0
	0.025	36	35	7
10	0.05	48	49	15
	0.1	52	58	28

It can be clearly seen that the lignin compound has good protease and lipase stabilising properties, even at very low concentrations.

Addition of the Ultrazine NA in soluble form results in even better enzyme stability.

Example 4

The formulation of Example 1 was prepared. Stabiliser (if any) was post dosed. The enzymatic activities in the liquid after 14 days of storage at 37°C was as follows when Ultrazine NA was added in a solution in 25% propyleneglycol in water:

25	% by weight	% Residual	% Residual
	Ultrazine NA	protease activity	lipase activity
		(with lipase)	(with protease
	0	13	4
	0.025	57	25
30	0.05	67	53
	0.1	70	63

The enzymatic activities in the liquid after 14 days of storage at 37°C was as follows when ultrazine NA was added in solid form:

	% by weight	% Residual protease activity (with lipase)	% Residual lipase activity (with protease)
	0	13	4
5	0.025	50	24
	0.05	62	53
	0.1	66	60

It can be clearly seen that the lignin compound has good protease and lipolase stabilising properties, even at very low concentrations. Addition of the Ultrazine NA in soluble form results in even better enzyme stability.

Example 5

The formulation of Example 3 was prepared. Various lignin compounds were added at a level of 0.1% by weight and the following stabilisation results were obtained, expressed as residual activity (in % of original activity) relative to the blanc (i.e. delta value).

		Delta % residual act.	
20	<u>Lignin compound</u>	<u>Lipase</u>	<u>Protease</u>
	Marasperse N-22 (ex Borregaard)	37	47
	Marasperse N-3 (ex Borregaard)	29	29
	Marasperse AG (ex Borregaard)	26	29
	Maracell (ex Borregaard)	46	50
25	Maracarb (ex Borregaard)	45	40
	Norlig 612 (ex Borregaard)	34	43
	Norlig (ex Borregaard)	38	45
	Ultrazine NA (ex Borregaard)	48	57
	Borresperse CA (ex Borregaard)	36	44
30	Borresperse NA	39	38
	Ultrazine CA (ex Borregaard)	44	50
	Ufoxane 2 (ex Borregaard)	30	29
	Ufoxane 3A (ex Borregaard)	38	35
	Na-lignosulphonate (ex Aldrich)	39	44

All lignins show lipase and protease stabilising effects.

CLAIMS

1. An enzymatic composition comprising from one or more
5 enzymes and one or more enzyme stabilisers, wherein the
stabiliser comprises a lignin compound and wherein the
composition is liquid.
2. Composition according to claim 1, wherein the enzyme is
10 selected from proteases, amylases, lipases and mixtures
thereof.
3. Composition according to claims 1-2 comprising from
0.0001 to 10% by weight of a lignin compound.
- 15 4. Composition according to claims 1-3, wherein the enzyme
comprises a protease.
5. Composition according to claims 1-4, further comprising
20 from 1 to 25 % by weight of electrolyte material.
6. Composition according to claims 1-5, further comprising
from 5 to 70% by weight of surfactant material.
- 25 7. A liquid enzymatic composition comprising from one or
more enzymes and one or more enzyme stabilisers,
characterised in that the stabiliser comprises a water-
soluble, cross-linked polymer containing sulphonate-groups.
- 30 8. Process of preparing an enzymatic composition by mixing
an enzyme preparation and one or more enzyme stabilisers,
wherein the enzyme stabiliser comprises lignin compounds.

9. Process according to claim 8, where in the lignin compound is brought in contact with the enzyme in a form in which the lignin is at least partially dissolved.
- 5 10. Process of mixing a lignin compound with an aqueous enzyme preparation or a non-aqueous surfactant containing enzyme preparation.

INTERNATIONAL SEARCH REPORT

Intern: J Application No
PCT/EP 96/02586A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 C11D3/386 C11D1/30

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 C11D C12N

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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A	WO 94 19529 A (VOLLMOND THOMAS ; LUND HENRIK (DK); TOFT ANNETTE HANNE) 1 September 1994 cited in the application see claims; example 5 ---	1
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☒ Further documents are listed in the continuation of box C.☒ Patent family members are listed in annex.

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INTERNATIONAL SEARCH REPORT

Interns 1 Application No
PCT/EP 96/02586

C/(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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